

**ESS Method 360.3:
Silica, Dissolved, Micro Level
Automated, Colorimetric**

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1.0 Application

- 1.1 This method may be used to determine concentrations of dissolved reactive silica in surface waters in the range from 0.05-2.00 mg SiO₂/L.
- 1.2 Approximately 25 samples per hour can be analyzed.

2.0 Summary of Method

Silica reacts with molybdate reagent in acid media to form a yellow silicomolybdate complex. This complex is reduced by ascorbic acid to form the molybdate blue color. The color intensity is proportional to the silica concentration.

3.0 Sample Handling and Preservation

Samples must be filtered through a 0.45 µm filter, cooled to 4 °C and analyzed within 28 days.

4.0 Interferences

- 4.1 Interference from phosphate, which forms a phosphomolybdate complex is eliminated by the oxalic acid introduced to the sample stream before the addition of the ascorbic acid reagent.
- 4.2 Tannin interference may also be eliminated by the addition of oxalic acid.
- 4.3 Hydrogen sulfide is an interference which must be removed by boiling an acidified sample before analysis.
- 4.4 Large amounts of iron and color may also interfere.

5.0 Apparatus

Technicon AutoAnalyzer II system consisting of:

- 5.1 Sampler IV with 30/h (6:1) Cam
- 5.2 Analytical Manifold
- 5.3 Proportioning Pump III
- 5.4 Colorimeter equipped with 2.0 x 50 mm flow cells

5.5 660 nm interference filters

5.6 Recorder/Printer

6.0 Reagents

6.1 Ammonium molybdate reagent: Dissolve 5 g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in 0.1 N sulfuric acid (2.8 mL concentrated sulfuric acid/L Milli-Q water) and dilute to 500 mL with the same. Store in an amber plastic container at 4°C. Stable for two months usually. If STDCAL value is higher than normal, make new.

6.2 Ascorbic acid reagent: Dissolve 8.8 g ascorbic acid in 250 mL Milli-Q water containing 25 mL acetone and dilute to 500 mL with Milli-Q water. Add 0.25 mL Levor IV solution. Store in an amber plastic container at 4°C.

6.3 Levor IV solution: Technicon No. 21-0332 or equivalent.

6.4 Oxalic acid solution: Dissolve 25 g oxalic acid in Milli-Q water and dilute to 500 mL. Store in a plastic bottle.

6.5 Milli-Q water: ASTM Type I reagent water, Millipore Corp., Bedford, MA.

6.6 Silica stock standard solution, 100 mg SiO_2 /L

6.6.1 Dilute 100 mL of Ricca or Banco 1000 mg/L standard solution (1 mL = 1.0 mg SiO_2) to 1L with Milli-Q water. (1 mL = 0.10 mg SiO_2)

6.6.2 Transfer the stock standard solution to a 1 L polyethylene bottle and store at 4°C.

6.7 Working standards (0.02-2.00 mg SiO_2 /L): Prepare the working standards by diluting the following volumes of stock standard solution (6.6) to the volume listed with Milli-Q water. Transfer the working standard solutions into polyethylene bottles and store at 4°C.

| Conc. mg SiO_2 /L | mL Stock Standard (6.6) |
|----------------------------|----------------------------|
| ----- | ----- |
| 0.02 | 0.20 mL/1L |
| 0.05 | 0.50 mL/1L |
| 0.15 | 1.50 mL/L |
| 0.20 | 0.20 mL/100 mL |
| 0.50 | 0.50 mL/100 mL |
| 1.00 | 1.00 mL/100 mL |
| 1.50 | 1.50 mL/100 mL |
| 2.00 | 2.00 mL/100 mL |

6.8 The 0.20 mg SiO_2 /L working standard is used for carryover correction.

7.0 Procedure

- 7.1 Set up the manifold as shown in Figure 1.
- 7.2 Allow the colorimeter, recorder and printer to warm up for 30 minutes. Obtain a stable baseline with all lines in Milli-Q water containing 0.5 mL LEVOR/500 mL. Then attach reagents, feeding Milli-Q water through the sample line.
- 7.3 Load sampler according to CFDA Tray Protocol.
- 7.4 Analyze according to procedures described in LIMS-CFDA Methods manual and General Auto Analyzer Procedures.

8.0 Calculations

The silica concentration is obtained directly from the LIMS plotter.

9.0 Precision and Accuracy

Precision and Accuracy data are available in the Inorganic Chemistry Unit Quality Assurance Manual.

10.0 References

- 10.1 Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, U.S. Geological Survey Techniques of Water-Resources Inv. Book #5 Ch. A1, p. 555 (1985).
- 10.2 Silicates in Water and Wastewater, Industrial Method No. 105-71W, Technicon Instruments Corporation, Tarrytown, NY (1973).

DISSOLVED SILICA

Range: 0.008-10 mg/L

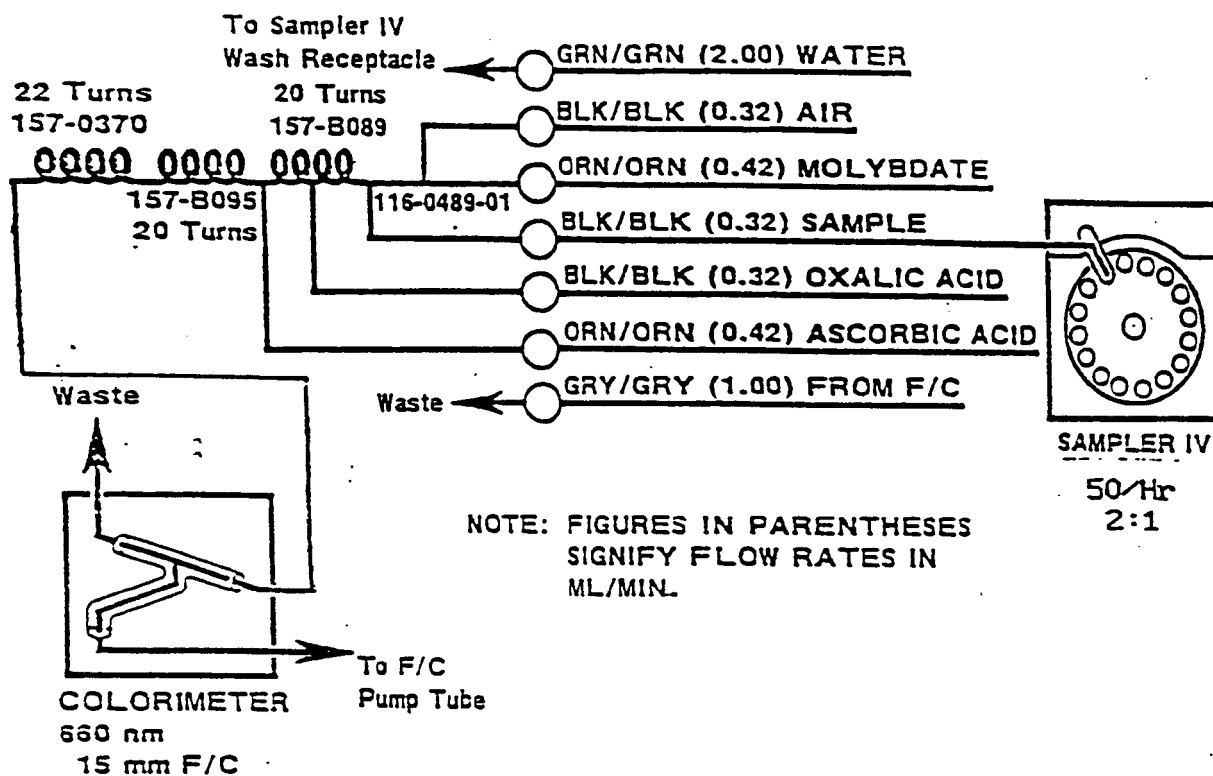


Figure 1. Manifold Set Up